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Monitoring the remediation of groundwater polluted by MSW landfill leachates by activated carbon and zeolite with spectral induced polarization technique

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ABSTRACT

Zeolite and activated carbon (AC) were used as the filling materials for a permeable reactive barrier (PRB) project in Tianziling Municipal Solid Waste (MSW) landfill in Hangzhou, China, for remediating groundwater contaminated by MSW leachates containing ammonium, chemical oxygen command (COD) and heavy metals (e.g. Fe^{3+}, Mn^{2+}). The spectral induced polarization (SIP), an emerging noninvasive geoelectrical method, was used to monitor the spatiotemporal contaminant distribution for its high sensitivity to pore fluid chemistry and mineral-fluid interface composition. Two columns filled with zeolite and AC were permeated with authentic groundwater collected from the landfill site, wherein the SIP responses were measured over a frequency range from 0.01 to 1000Hz. The adsorption capacities of zeolite for COD (7.08mg/g) and ammonium (9.15mg/g) were higher than those of AC for COD (2.75 mg/g) and ammonium (1.68 mg/g). The SIP responses were fitted by Cole-Cole model to obtain the relaxation time normalized chargeability (m_n). The m_n of zeolite were positively linear with the adsorbed ammonium (R^2=0.999) and COD (R^2=0.999); while the m_n of AC were negatively linear with the adsorbed ammonium (R^2=0.9341) and COD (R^2=0.9790). These results suggest that the m_n could be a surrogate for the contaminant saturation. Therefore, the laboratory real-time noninvasive SIP for zeolite and AC showed promises for monitoring the saturation process, providing the basis for the future field PRB monitoring.

Keywords: spectral induced polarization, MSW landfill groundwater, activated carbon, zeolite, adsorption.

1 INTRODUCTION

Landfill is the most widely used method for municipal solid waste (MSW) disposal in China, USA, and other developing countries(Han et al., 2016; Ye et al., 2019). There are approximately 1600 regulated MSW landfills and over 27000 unregulated MSW waster dump sites in China which may cause serious groundwater pollution due to the leachate leakage to the downstream groundwater(Ye et al., 2019). The remediation of sites contaminated by the MSW landfill leachates is an emerging global challenge for its complex constituents which mainly includes ammonium (NH_4^+), organic matter such as chemical oxygen demand (COD) and heavy metals(Han et al., 2016; Ye et al., 2019). The permeable reactive barrier (PRB) is one of the most effective in-situ remediation technologies, which was usually constructed across the groundwater flow path with high immobilization ability and permeability(Chen et al., 2022; Ye et al., 2019; Zhang et al., 2022). For the groundwater contaminated by the MSW leachate with high concentration of NH_4^+ and COD, it is challengeable to remediate the complex contaminant by single reactive medium and the multi-layer PRBs were generated for complex contaminants remediation(Faisal et al., 2020; Kong et al., 2015; Kumarasinghe et al., 2018; Pawluk & Fronczyk, 2015). Zeolite was widely used as PRB reactive medium for NH_4^+ remediation in groundwater due to its high cation exchange capacity(Chen et al., 2022; Zhang et al., 2022) and the activated carbon (AC) was effective at remediating organic matters from groundwater for its high specific surface area, oxygen-containing functional groups and π-π interaction between aromatic rings of AC and organic matters (Ben Moshe & Furman, 2022; Mohan et al., 2008; Wong et al., 2018).

Monitoring PRB performance is of great significance because of the deterioration caused by adsorbent saturation(Ye et al., 2019). However, the existing monitoring methods for PRBs mostly depended on sensors, sampling from the monitoring wells installed intrusively in the PRBs and laboratory chemical analysis which suffered from expensive testing costs, delayed analysis results as well as flow field disturbance by monitoring wells and sampling (Chen et al., 2022; Hao, Cao, et al., 2021; Q. Wang et al., 2022). The spectral induced polarization (SIP) is an emerging geo-electrical real-time and nonintrusive monitoring method for contaminant remediation, involving the alternating current injection ranging from 0.01-1000 Hz and potential measurement (Ben Moshe & Furman, 2022; Hao, Cao, et al., 2021). Compared with the direct current (DC) conductivity tomography technique providing the bulk DC conductivity of the mixture of pore fluid and porous matrix without further distinction, the SIP can provide complex conductivity where the real conductivity is sensitive to fluid chemistry and the imaginary conductivity is sensitive to the constituent of electrical double layer (EDL) near the solid-liquid interface, and therefore the SIP method can provide detailed information to distinguish between porous matrix and pore fluid which makes it possible to monitor contaminant saturation (Ben Moshe & Furman, 2022; Hao, Cao, et al., 2021).

A few studies reported the SIP signature of AC and zeolite remediating contaminants including organic matters and inorganic cations (e.g. NH_4^+ and heavy metals) (Bate et al., 2022; Ben Moshe & Furman, 2022; Hao, Cao, et al., 2021). Bate et al. (2022) monitored the SIP responses of zeolite when permeated by synthetic Pb^{2+}, NH_4^+ and COD solution. All the imaginary conductivities decreased with adsorbed contaminant accumulation. Hao et al. (2021) observed remediation process of Pb^{2+} by AC and a new peak appeared with the precipitation of Pb-hydroxide and the magnitude of imaginary conductivity increased with Pb^{2+} adsorption. The process of organic dye (crystal violet) remediated by AC was monitored using SIP by Ben Moshe and Furman (2022), the magnitude of original imaginary peak decreased and a new peak appeared, attributing to the movement of dye molecules that were attached to AC surface through π-π interaction. While most of SIP researches about zeolite and AC monitoring used the synthetic solution with single contaminant, few researches reported the SIP monitoring of zeolite and AC permeated by authentic multi-contaminant polluted water. Kirmizakis et al. (2020) remediated the olive oil mill wastewater with high phenols concentration by biochar and reported that the imaginary conductivity of SIP increased with phenols uptake. To the best of our knowledge, no research reported the SIP monitoring process of PRB materials permeated by authentic groundwater polluted by MSW leachate of high NH_4^+ and COD concentrations.

In this study, the permeation of the columns filled with PRB materials, namely zeolite and AC, by the authentic groundwater collected from downstream of a MSW landfill were monitored by spectral induced polarization. The purposes of this study were to monitor the pollution remediation noninvasively with SIP, and provide basis for the future filed SIP monitoring for PRBs.

2 MATERIALS AND METHODOLOGY

Natural clinoptilolite zeolite (Jinyun county, Zhejiang Province, China) and commercial activated carbon (AC) (Shanghai Xinhui Co., Ltd, China), manufactured from coconut husk after pyrolysis at 700℃ and vapor activation at 300℃, with the porosity of 0.6 and grain size of 1-2 mm were used. The bulk density of zeolite and AC were 1.3 and 0.63 g/cm^3, respectively. Both the zeolite and AC were rinsed thoroughly and soaked in deionized water (DI water) over 24 hours to remove the impurities, and were then oven-dried at 105℃ for 24 hours before use. The specific surface areas of zeolite and AC from nitrogen adsorption-desorption test (Autosorb-1-C, Quantachrome) were 7.975 m^2/g and 1001.786 m^2/g.

The concentrations of NH_4^+, chemical oxygen demand (COD) and heavy metals (e.g. Fe^{3+} and Mn^{2+}) of authentic groundwater (AG) sampled downstream of the Tianziling MSW landfill (Hangzhou, China) were analysed by nesslerization method, colorimetric method and ion chromatography, respectively. Column breakthrough tests for the permeation of AG through zeolite and AC were performed, respectively. Zeolite and AC were compacted in an acrylic column (21.5 cm×4 cm, height×inner diameter) filled with DI water to increase water saturation. The AG was pumped from column bottom at 2 mL/min peristaltically and the time required for 1 pore volume (PV) to flow through was about 81 minutes. The effluent was collected from the column top by automatic fraction collector at time intervals of 10 mins, and was then analysed for chemical speciation.

Spectral induced polarization (SIP) measurements were performed at frequency range from 0.01 to 1000 Hz (PSIP instrument, Ontash &Ermac, Inc., NJ, USA). Silver rods coated with silver chloride were used as current and potential electrodes. A pair of spiral current electrodes were placed at either side of the specimen, while 2 pairs of potential electrodes were placed at both the inflow and outflow side with spacing of 5 cm and an average distance of 5 cm from the adjacent current electrode (as shown in Figure 1). The SIP signals can be expressed as complex conductivity (σ^*), which can be calculated from the measured magnitude ($|\sigma|$) and phase shift (φ) (Hao, Cao, et al., 2021):

$$\begin{aligned} \sigma^* &= |\sigma| e^{i\varphi} = \sigma' + i\sigma'' \\ \sigma' &= |\sigma| \cos(\varphi) \\ \sigma'' &= |\sigma| \sin(\varphi) \end{aligned} \tag{1}$$

where σ' is real conductivity, representing the conductance and energy loss; σ'' is imaginary conductivity, representing charge polarization and energy storage; and $\mathrm{i} = \sqrt{-1}$.

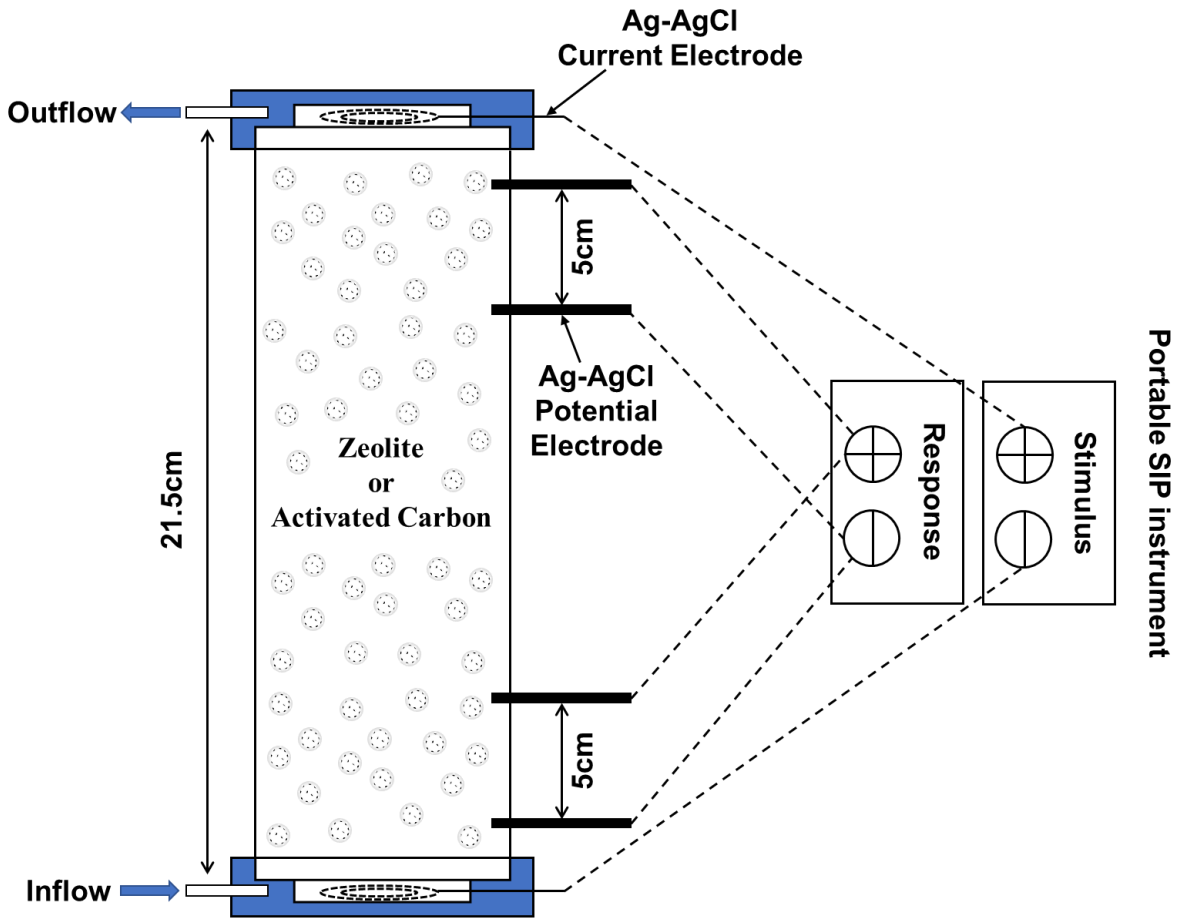


Figure 1. *Diagram of the experimental SIP setup for the column breakthrough tests.*

The phenomenological Cole-Cole model was widely used to fit and analyze the complex conductivity with single relaxation peak. Expressions of σ^* in Cole-Cole space are(Hao, Ye, et al., 2021):

$$\sigma^*(\omega) = \sigma_\infty \left(1 - \frac{m}{1 + (i\omega\tau)^c}\right) \tag{2}$$

$$m = \frac{\sigma_\infty - \sigma_0}{\sigma_\infty} \tag{3}$$

where σ_∞ and σ_0 represent the upper and lower limits of the conductivity at high (after current induced polarization) and low (before current induced polarization) frequencies, respectively; m is the chargeability, relating to the magnitude of polarization(Mellage et al., 2022); c is the shape exponent

typically ranging from 0.1 to 0.6 (Slater et al., 2006); $\omega = 2\pi f$ is the angular frequency; τ is the relaxation time. In addition, normalized chargeability m_n ($= m \times \sigma_0$) was often used to facilitate data comparison when fluid conductivity changed(Bate et al., 2020; Wu & Peruzzo, 2020).

3 RESULTS AND DISCUSSION

3.1 Chemical composition of authentic groundwater

Tianziling Landfill which located in Hangzhou, China started operation in 1991 for primary municipal solid wastes, and closed in 2020. The Phase Ⅰ of the landfill was in operation from 1991 to 2007, when the landfill regulation on liner was not required, and leachate might penetrate and contaminate downstream ground. Phase Ⅱ of the landfill was in service from 2007 to 2020, where liner was applied in accordance to GB/T 51403, and leachate leaking was prevented. The old MSW leachate is characterized by its high concentration of NH_4^+ and COD with low ratio of BOD/COD (<0.1) (Kjeldsen et al., 2002; Renou et al., 2008). After as long as 30 years of operation, NH_4^+ concentration increased due to anaerobic degration of MSW, COD decreased due to the completion of fresh MSW over time. The COD in the old leachate which could not be biodegradable was attributed to refractory organic molecules like humic acid and fulvic acid with high molecule weight consisting of many aromatic rings, carboxyl, alcoholic and phenolic hydroxyl (Albaiges et al., 1986; Halim et al., 2012; Kjeldsen et al., 2002; Renou et al., 2008; S. Wang et al., 2006, 2008). The main composition of AG (Table 1) were NH_4^+, COD and heavy metals (i.e. Fe^{3+}, Mn^{2+}), all of which exceeded the limits of the groundwater quality, namely NH_4^+ < 10 mg/L, COD < 10mg/L, and Mn < 1.5mg/L (GB/T 14848-2017).

Table 1. *Chemical composition of authentic groundwater from Tianziling Landfill*

Content	pH	Conductivity mS/cm	NH_4^+ mg/L	COD_{Mn} mg/L	Fe^{3+} mg/L	$Mn2^+$ mg/L	Na^+ mg/L	K^+ mg/L	Ca^{2+} mg/L
Concentration	7.7-7.9	3.48-4.77	142-313	80-140	0.79	2.65	234-290	125-159	47.8-60.2

3.2 Spectral induced polarization responses

3.2.1 Zeolite

3.2.1.1 Real conductivities (σ')

The temporal evolutions of real conductivities (σ') were shown in Figure 2. Before AG permeation (0PV, zeolite saturated with DI water), σ' at influent and effluent sides of zeolite column were at the value of 50 μS/cm (Figure 2.a, b). For the effluent side at 18PV (Figure 2.a), σ' increased from 50 to 1100 μS/cm due to replacement of DI water by AG. From 18PV to 167PV, σ' increased gradually from 1100 to 1300 μS/cm, attributing to the contaminant breakthrough. The σ'evolution of influent side was similar to that of effluent side, while it reached peak value faster due to earlier arrival of AG front.

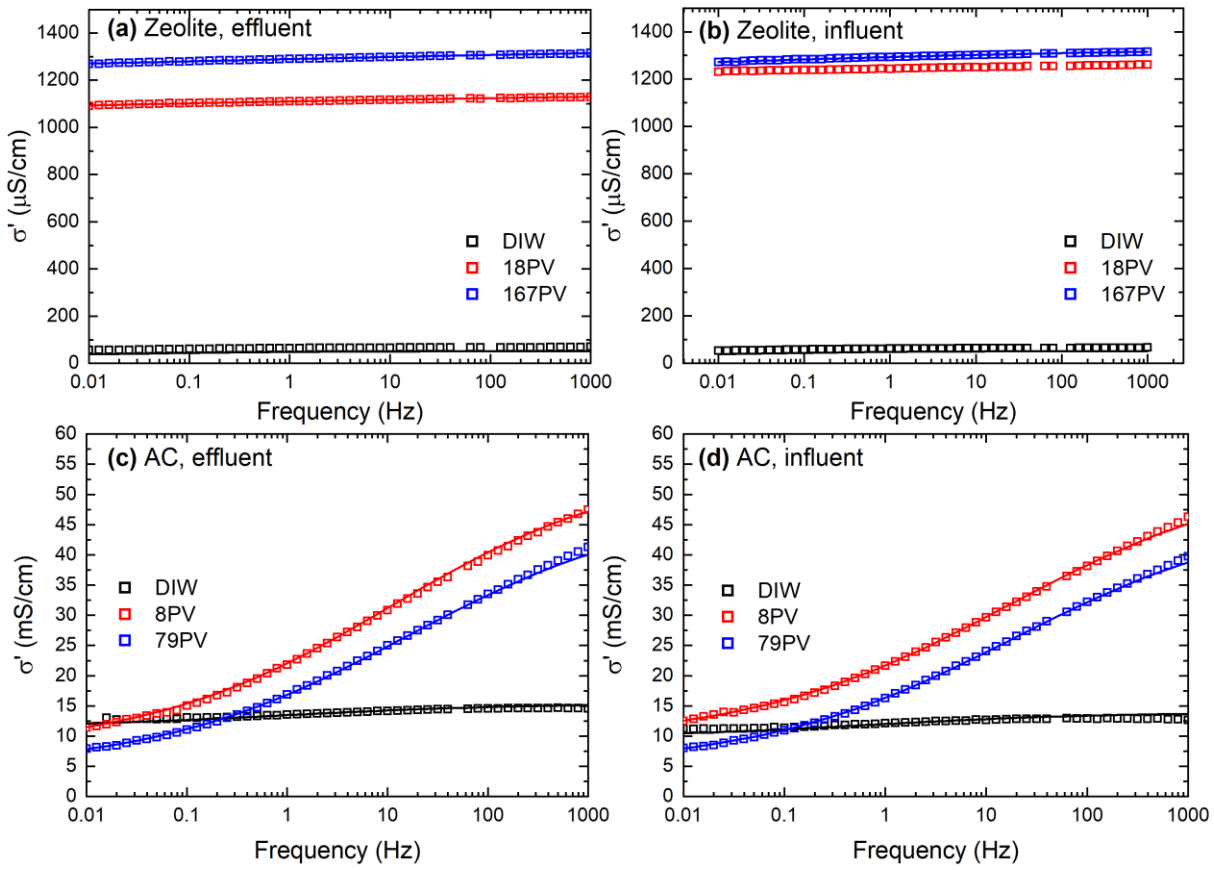


Figure 2. *Temporal evolution of real conductivities (σ') and Cole-Cole fits of: (a) effluent side and (b) influent side of zeolite, (c) effluent side and (d) influent side of AC.*

3.2.1.2 Imaginary conductivity (σ'')

The temporal evolutions of imaginary conductivities (σ'') were shown in Figure 3. Before AG permeation (0PV, saturated with DI water), σ'' at influent and effluent sides of zeolite column were smooth with a clear peak at 0.04 Hz of 3.5 µS/cm (Figure 3. a,b). At influent side, after AG permeation 18PV, σ'' at 0.04 Hz increased from 3.5 to 6.3 µS/cm and σ'' fluctuated severely due to multicomponent contaminants of AG, meanwhile, the spectrum became flat (Figure 3. b). Suppression of the zeolite σ'' by NH4+ adsorption has been demonstrated by previous research. However, in the range of 0 to 18 PV, the increase of σ'' at 0.04 Hz may be attributed to the adsorption of COD, which consists of numerous polarizable groups such as hydroxyl, carboxyl, and phenyl groups. (Bate et al., 2022). Similar conclusion has been drawn by Mellage et al. who found σ'' of sandy loam increased with content of solid organic matter due to the increasing polarizable sites (Mellage et al., 2022).From 18 to 167PV, σ'' increased from 6.4 to 8.1 µS/cm and remained at 8.1 µS/cm after 167PV, attributing to the accumulation of contaminants and saturation of adsorption sites. The σ'' evolution at effluent side was similar to that at influent side,

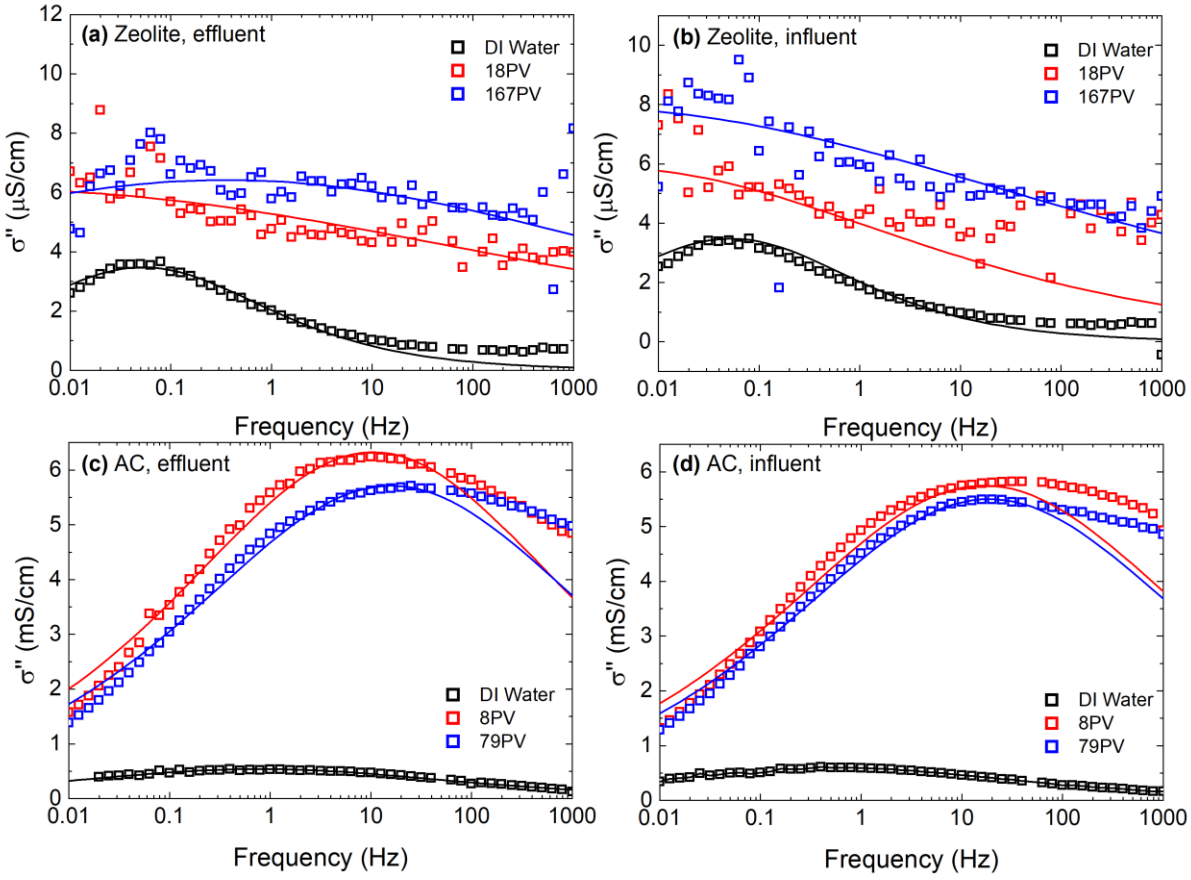


Figure 3. *Temporal evolution of imaginary conductivities (σ") and Cole-Cole fits of: (a) effluent side and (b) influent side of zeolite, (c) effluent side and (d) influent side of AC.*

3.2.1.3 Normalized chargeability (m_n)

The Cole-Cole model (Equation.2) was used to fit the SIP spectrum by pyGIMLi code (Rücker et al., 2017) (Figure 2,3). The normalized chargeability ($m_n = m \times \sigma_0$) evolved with permeation due to the contaminant adsorption (Figure 4. a). As shown in Figure 4. a, both the adsorbed NH_4^+ and COD concentrations were positive linear with the m_n of zeolite. Because the normalized chargeability quantifies the magnitude of induced polarization, the positive linear relationship between the m_n and adsorbed contaminant concentrations suggested the net SIP responses increased attributable to the polarizable groups in organic matters (stimulated the induced polarization) and the NH_4^+ (suppressed the induced polarization of zeolite) (Bate et al., 2022; Elis et al., 2016; Mellage et al., 2022). The linear relationships between the adsorbed contaminant concentrations and m_n indicated that m_n could be a surrogate as the contaminant saturation in zeolite.

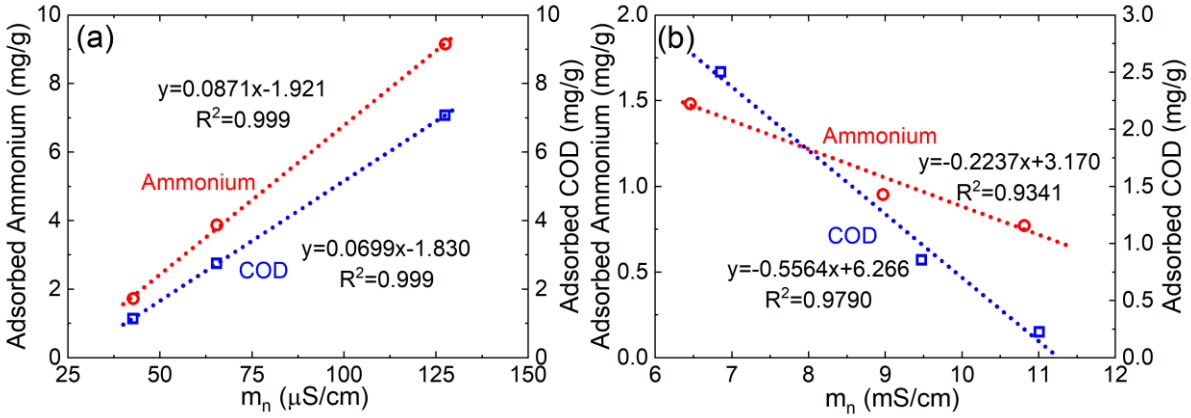


Figure 4. *Relationship between the normalized chargeability (m_n) and the adsorbed contaminant for: (a) zeolite, (b) AC*

3.2.2 Activated cation

3.2.2.1 Real conductivities (σ')

Before AG permeation (0PV, AC saturated with DI water), σ' of AC column were 10 mS/cm (Figure 2. c, d). For the influent side at 8PV (Figure 2. c), σ' at 1000 Hz increased from 10 to 46 mS/cm due to pore water conductivity increase attributable to replacement of DI water by AG. From 8 to 79PV, σ' of all frequency decreased about 5 mS/cm. σ' reached the peak values as NH_4^+ breakthrough, while as COD adsorbed by AC, σ' decreased and stayed steady when COD breakthrough. The σ' at effluent side was similar to that of influent side where σ' increased to peak value as NH_4^+ breakthrough and decreased to steady value as COD breakthrough.

3.2.2.2 Imaginary conductivity (σ'')

Before AG permeation (0PV, saturated with DI water), σ'' at influent and effluent sides of AC column were smooth with a clear peak at 1 Hz of 0.6 mS/cm. At influent side, after AG permeation 8PV, σ'' at 1 Hz increased from 0.6 to 4.9 mS/cm due to the DI water replaced by AG with high conductivity and the peak frequency shifted from 1 Hz to 16 Hz and the spectrum became steep (Figure 3. d). From 8 to 79 PV, the peak σ'' values decreased from 5.8 to 5.5 mS/cm with the peak frequency shifting from 16 Hz to 20 Hz. While as the NH_4^+ breakthrough at 8PV (reached adsorption capacity), the continuing decreasing σ'' should be attributed to the adsorbed COD and σ'' stayed steady after COD breakthrough (79PV). A similar decreased σ'' at peak frequency was reported by Ben Moshe et at. who remediated charged organic dye by AC (Ben Moshe & Furman, 2022). It was well acknowledged that the changes of σ'' spectrum stemmed from the change of diffusion coefficients of the ions constituting the EDL (Ben Moshe & Furman, 2022; Hao, Cao, et al., 2021; Hao, Ye, et al., 2021). The decreased σ'' at the peak frequency ranging from 10 Hz to 20 Hz was attributed to the lower diffusion coefficients ($1.25\times10^{-11}\ m^2/s$) of adsorbed organic matters with high molecule weight around the EDL of AC compared to the smaller cations with higher diffusion coefficients (e.g. Na^+, $1.33\times10^{-9}\ m^2/s$) which occupied the EDL previously (Ben Moshe & Furman, 2022; Hao et al., 2015; Schwartz & Furman, 2012). It has been discussed that the main mechanisms for the COD adsorption by AC were π-π interactions together with electrostatic attractions and H-bonds (Ben Moshe & Furman, 2022; Gupta & Khatri, 2019). Because of the very high polarizing frequency of π-π system (from megahertz to terahertz), the polarization mechanism from 10 to 20 Hz could be attributed to the movement of the polarizable group (e.g. hydroxyl, carboxyl and phenyl groups) of organic molecules along the direction of the alternating-electric field (Ben Moshe & Furman, 2022; Busl et al., 2012). σ'' at effluent side was similar to that of influent side. Therefore, the increase of σ'' could surrogate the saturation of NH_4^+ while the decrease of σ'' can be regarded as the proxy for the saturation of COD.

3.2.2.3 Normalized chargeability (m_n)

Negative linear relationship between adsorbed contaminant concentrations and m_n of AC was observed (Figure 4. b). It could be observed from Figure 4 that the m_n of AC (mS/cm) is about 2 orders of magnitude higher than that of zeolite (μS/cm). Due to the adsorbed contaminants (NH_4^+ and COD) with lower diffusion coefficients were harder to be polarized than the naturally occurring cations within EDL (e.g. Na^+ and K^+), the magnitude of induced polarization decreased with adsorbed contaminants, resulting in the negative linear relationships between m_n and adsorbed NH_4^+ and COD whose relationships were inverse with those of zeolite. Therefore, m_n could be regarded as proxy of contaminant saturation of AC.

4 FUTURE OUTLOOK

As the composition of groundwater contaminated by MSW landfill leachates is too complex, more efforts will be required in the future to obtain detailed information on the composition of contaminated groundwater. This is necessary to fully understand the meaning of SIP signals and apply the SIP

technique to the monitoring of field barriers. The conclusions of this study were based on the laboratory experiments. In the future, the obtained conclusions should be verified in the field experiments.

5 CONCLUSIONS

In this study, spectral induced polarization was used to monitor the permeation of zeolite and activated carbon with the authentic MSW landfill groundwater with high concentration of NH_4^+ and COD. The normalized chargeability (m_n) of zeolite was positively linear with the adsorbed NH_4^+ (R^2=0.999) and COD (R^2=0.999) concentrations while that of AC was negatively linear with the adsorbed NH_4^+ (R^2=0.9341) and COD (R^2=0.9790) concentrations. The good relationship between SIP responses and contaminant saturation provided basis for field nonintrusive monitoring of PRB filled with zeolite and AC by SIP in the future.

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